

## UNITED STATES PATENT OFFICE.

ROBERT C. MORAN, OF WOODBURY, NEW JERSEY, ASSIGNOR TO E. I. DU PONT DE NEMOURS & COMPANY, OF WILMINGTON, DELAWARE, A CORPORATION OF DELAWARE.

## ACCELERATION OF GELATINIZATION OF CELLULOSE NITRATE.

No Drawing.

Application filed June 19, 1924. Serial No. 721,004.

This invention relates to the acceleration of the gelatinization of mixtures of cellulose nitric esters with liquid nitric esters, particularly, glyceryl trinitrate and its mixtures with freezing-point depressants such as nitrated diglycerine, nitrated sugars, etc.

In the manufacture of non-solvent gelatinized nitro-glycerine explosives, such as blasting gelatine and gelatine dynamite, the mixture of nitrocellulose and nitroglycerine is gelatinized previous to the addition of the rest of the ingredients. The rate of gelatinization of such a mixture is dependent for the most part upon three factors; first, the temperature at which the gelatinization is effected, second, the quantity and quality of the nitrocellulose used, and, third, whether the nitroglycerine is straight glyceryl trinitrate or a mixture thereof with a freezing-point depressant. Each of these factors requires careful control measures in order to insure satisfactory rapidity of gelatinization.

I have discovered that the rate of gelatinization of cellulose nitric esters with liquid nitric esters, particularly glyceryl trinitrate and its mixtures with freezing-point depressants, can be accelerated by means of small amounts of aliphatic alcohols which are soluble in glyceryl trinitrate. This discovery makes it possible to exert a more or less arbitrary control over the rate of gelatinization of such mixtures, to shorten the time of manufacture and to overcome one of the difficulties encountered in the manufacture of low freezing gelatine dynamites containing for example nitrated polymerized glycerine as a freezing-point depressant of trinitroglycerine.

As representative accelerators of the monohydric alcohol class, I mention methyl and ethyl alcohols, and as examples of accelerators of the polyhydric alcohol class there may be mentioned ethylene and trimethylene glycols. I prefer to use the dihydric alcohols, and particularly the lower glycols, that is the glycols having from two to four carbon atoms, (1) because of their solubility values in the glyceryl trinitrate or other liquid nitric ester which is to be gelatinized with the nitrocellulose, and (2) because of their low volatility as compared with methyl and ethyl alcohols. The butylene glycols (for instance the alpha or

beta isomers) are capable of effecting an acceleration of the same order as that of the other glycols above mentioned. Glycerol, apparently because of its low solubility in glyceryl trinitrate, exerts little, if any accelerating action on the gelatinization of a mixture of glyceryl trinitrate and nitrocellulose.

The quantity of the above accelerators required to bring about a decided effect in the rate of gelatinization of mixtures of cellulose nitric esters with liquid nitric esters as, for instance, glyceryl trinitrate, straight or in mixture with freezing-point depressants, is very small. For example, 1% ethyl alcohol, when added to a mixture of 8% nitrocellulose and 92% low-freezing nitroglycerine, while maintaining the resulting mixture at a temperature between 100° F. and 125° F. reduced the time required for gelatinization from 12 minutes to 5½ minutes; 1% ethylene glycol reduced the time from 12 minutes to 5¾ minutes; and 1% trimethylene glycol from 12 minutes to 6 minutes, other conditions remaining the same. While the proportion of accelerator used may be 1% or more based upon the weight of liquid nitric ester to be gelatinized, I prefer to use substantially less than 1% of the accelerator—for example from about 0.1% to 0.6%.

In carrying out my process, the accelerator may be either added to the mixture of cellulose nitrate with the liquid nitric ester in the mixing-bowl, or previously incorporated in the liquid nitric ester component.

When gelatinization of the liquid nitric ester and nitrocellulose is completed, the other gelatin dynamite ingredients may be introduced in the customary way. These other ingredients are usually an oxygen-carrying inorganic salt such as sodium or ammonium nitrate, a combustible non-explosive material such as corn meal or wood-meal, and in certain cases various other substances such as nitrohydrocarbons, sulphur, chalk, sodium chloride, etc., depending upon the kind of blasting operation for which the high explosive is being made.

I claim:

1. The process of accelerating the gelatinization of cellulose nitrate with a liquid nitric ester which comprises mixing said substances in the presence of an aliphatic

alcohol that has a greater solubility in said liquid nitric ester than has glycerol, the quantity of said alcohol being sufficiently small so as to produce substantially no effect upon the sensitiveness of the mixture as an explosive.

2. The process of accelerating the gelatinization of cellulose nitrate with a liquid nitric ester which comprises mixing said substances in the presence of a polyhydric aliphatic alcohol that has a greater solubility in said liquid nitric ester than has glycerol.

3. The process of accelerating the gelatinization of cellulose nitrate with a liquid nitric ester which comprises mixing said substances in the presence of a polyhydric alcohol containing from two to four carbon atoms that has a greater solubility in said liquid nitric ester than has glycerol.

4. The process of accelerating the gelatinization of cellulose nitrate with a liquid nitric ester which comprises mixing said substances in the presence of a dihydric alcohol containing from two to four carbon atoms.

5. The process of accelerating the gelatinization of cellulose nitrate with a liquid nitric ester which comprises mixing said substances in the presence of trimethylene glycol.

6. A process as set forth in claim 1 in which the proportion of alcohol is not more than about 1% based on the weight of liquid nitric ester present.

7. A process as set forth in claim 2 in which the proportion of alcohol is not more than about 1% based on the weight of liquid nitric ester present.

8. A process as set forth in claim 3 in which the proportion of alcohol is not more than about 1% based on the weight of liquid nitric ester present.

9. A process as set forth in claim 4 in which the proportion of alcohol is not more than about 1% based on the weight of liquid nitric ester present.

10. A process as set forth in claim 5 in which the proportion of glycol used is not more than about 1% based on the weight of liquid nitric ester present.

11. A process as set forth in claim 1 in which the liquid nitric ester comprises glyceryl trinitrate.

12. A process as set forth in claim 1 in

which the liquid nitric ester comprises glyceryl trinitrate and a freezing point depressant.

13. A process as set forth in claim 3 in which the liquid nitric ester comprises glyceryl trinitrate.

14. A process as set forth in claim 3 in which the liquid nitric ester comprises glyceryl trinitrate and the nitrated derivative of a polyhydroxy aliphatic compound containing more than three hydroxyl groups.

15. A process as set forth in claim 4 in which the liquid nitric ester comprises glyceryl trinitrate and a freezing-point depressant.

16. The process of making a high explosive composition which comprises gelatinizing a mixture of cellulose nitrate, nitroglycerine and a freezing-point depressant in the presence of an aliphatic alcohol which is more soluble in nitroglycerine than in glycerol, and then adding to the gelatinized mixture the other ingredients required in the finished explosive composition, the quantity of said alcohol being sufficiently small so as to produce substantially no effect upon the sensitiveness of the mixture as an explosive.

17. A high explosive composition comprising cellulose nitrate gelatinized with a liquid nitric ester and containing an aliphatic alcohol which is more soluble in the liquid nitric ester than is glycerol, the quantity of said alcohol being sufficiently small so as to produce substantially no effect upon the sensitiveness of the mixture as an explosive.

18. A process as set forth in claim 16 in which the alcohol is a glycol having between one and five carbon atoms.

19. A composition as set forth in claim 17 in which the alcohol is a polyhydric alcohol containing from two to four carbon atoms.

20. A high explosive composition comprising a gelatinized mixture of cellulose nitrate and nitroglycerine and containing a glycol having from two to four carbon atoms.

21. A high explosive composition comprising a gelatinized mixture of cellulose nitrate and nitroglycerine and containing not more than 1% of a glycol having from two to four carbon atoms.

In testimony whereof I affix my signature.

ROBERT C. MORAN.